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LUMINESCENCE OF $PbWO_4$ SINGLE CRYSTALS DOPED WITH FLUORINE

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The influence of fluorine doping on the luminescent properties of lead tungstate, $PbWO_4$, a scintillating material used in high-energy physics, was studied. Two series of crystals that were grown by the Czochralski method in two different laboratories were investigated. It was shown that the luminescent properties of the fluorine-doped samples were similar although those of the undoped $PbWO_4$ samples from the different series differed significantly. It was concluded that this was associated with the formation of WO_3F complexes in the fluorine-doped $PbWO_4$ crystals.

Keywords: *lead tungstate, fluorine doping, luminescent properties, thermoluminescence, WO_3F complexes.*

Introduction. Lead tungstate ($PbWO_4$) is used as a scintillation detector in high-energy physics (CMS, ALICE, PANDA projects) [1, 2]. Future prospects for application of $PbWO_4$ scintillators, e.g., as active protection for experiments on the recording of rare events at ultralow temperatures [3] and as detectors for the separation of scintillation and Cherenkov radiation [4], are under discussion.

The prospects of using $PbWO_4$ are limited significantly by the relatively low light output at room temperature (200 photons/MeV). This is associated with intracenter thermal luminescence quenching. Efforts in at least two directions are currently being made in order to improve this crystal parameter. A relatively small temperature decrease (to $-25^{o}C$) is known to be able to increase the light output by several times. However, the luminescence decay time increases noticeably, the radiation stability of the crystals decreases, and the time for restoring defects after irradiation increases as the light output increases [5].

Another possibility for improving the light output is to dope $PbWO_4$ crystals with various impurities. The ability to dope $PbWO_4$ with negatively charged ions (F, Cl, I, S) is very interesting. The results indicated that doping by F^- had the greatest effect on the optical and spectral characteristics of $PbWO_4$ [6]. However, reported results on the luminescent properties of $PbWO_4$:F differed markedly [7–10]. It should be noted that the overwhelming majority of results on fluorine-doping of $PbWO_4$ was obtained on crystals grown by the Bridgman method. It is thought that the Bridgman method with a closed crucible is more suitable for such doping of $PbWO_4$ because the probability of F vaporizing during the growth process is reduced [11]. However, crystals grown by the Czochralski method are mainly used at present for practical application. It is noteworthy that production of $PbWO_4$:F crystals by the Czochralski method can be difficult because of the low segregation coefficients of F in this growth method, as previously suggested [8]. Nevertheless, it was shown in several studies [12, 13] that fluorine-doping improves the radiation stability and luminescent properties of $PbWO_4$ crystals grown namely by the Czochralski method.

Herein the effect of an F dopant on the luminescent properties of $PbWO_4$ grown by the Czochralski method is studied. Two series of samples prepared in different laboratories are investigated.

Experimental. Luminescence spectra with UV and VUV excitation in addition to thermoluminescence (TL) spectra and luminescence decay kinetics were measured on a Superlumi apparatus situated in the synchrotron channel

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of the DORIS III storage ring (DESY, Hamburg, Germany) [14]. The primary monochromator enabled the sample to be irradiated by photons in the energy range 3.7–30.0 eV. Luminescence spectra were measured using an ARC SpectroPro SP-308 monochromator operating in spectrograph mode and were normalized to the spectral sensitivity function of the recording system. The sample was placed in a He flow cryostat in order to take measurements in the temperature range 10–300 K. All measurements were taken from the surface of fresh crystal chips.

The studied crystals were grown by the Czochralski method at the Institute for Single Crystals, Kharkiv, Ukraine (series K) and at I. Franko Lviv National University, Ukraine (series L). Both series incorporated nominally pure $PbWO_4$ and fluorine-doped $PbWO_4$:PbF_2.

The charge for series K crystals was synthesized by a solid-state method using PbO (ultrahigh purity) and WO_3 (ultrahigh purity). The activator was added to the charge as PbF_2 (ultrahigh purity) at a melt concentration of 28,000 ppm. Crystals were grown in an inert atmosphere from Pt crucibles along the [001] direction using an Analog apparatus. The resulting crystals (≤25 mm diameter and ≤50 mm length) were typically transparent and did not contain macroscopic inclusions.

Series L crystals were grown in an inert medium in a Pt crucible on a Donets apparatus. Starting materials PbO and WO_3 (99.99%) were mixed in a 1:1 ratio. The PbF_2 melt concentration was 4000 ppm. The resulting crystals (≤15 mm diameter and ≤50 mm length) were transparent.

The dopant composition of the $PbWO_4$ samples was determined using spark mass spectrometry. According to the analytical results, the F concentration in the doped samples was ≈10 ppm. An uncontrolled impurity of Y (240 ppm) was also detected in the series K sample. The concentration of this impurity in the series L sample was 0.15 ppm. Furthermore, impurities of Ca (~10 ppm) and Mo (0.9 ppm) were found in the series L sample. The concentrations of other impurities (from Li to U) in the samples were less than 0.4 ppm (typically ppm). The results confirmed the suggestion [8] about the low (<<1) segregation coefficient of F into the $PbWO_4$ crystal structure.

Results and Discussion. *Luminescence spectra of $PbWO_4$.* The luminescence spectrum of pure $PbWO_4$ is known to be characterized by at least two bands with maxima in the blue (420–460 nm, blue band) and green (480–520 nm, green band) spectral regions [15] (a review and references therein). It was shown in numerous studies that the blue band is fundamental and due to radiative relaxation of excitons autolocalized on WO_4^{2-} complexes. The green band is due to emission at WO_3-type (oxygen vacancy) defects in the crystal structure [15, 16].

Figure 1 shows the temperature dependences of the luminescence spectra of both pure $PbWO_4$ and fluorine-doped samples. It is noteworthy that the luminescence excitation energy (13.7 eV) is known to exceed the crystal band gap (4.6 eV) [17]. This energy is capable of creating separated electron–hole pairs that can further be captured by centers responsible for the blue and green luminescence bands. Table 1 presents the position of the maxima and full-width at half-maximum (FWHM) of the emission bands at several temperatures.

The blue emission band with a slight contribution from the green band dominated for the series K pure $PbWO_4$ crystal over the whole temperature range (Fig. 1a). The contribution of the latter was observed as a low-energy shoulder. It is noteworthy that the domination of the blue component in the luminescence spectrum was indicative of a low concentration of structural defects and high structural perfection of the series K $PbWO_4$ crystal lattice.

The luminescence spectrum of the pure series L $PbWO_4$ crystal (Fig. 1b) at temperatures 200 K was characterized by a distinct green band. The spectrum shifted to the high-energy region and the intrinsic blue luminescence began to dominate as the temperature was decreased further. The observed shift of the luminescence maximum was related to a change of the relative contributions to the spectrum of the two component bands.

Doping the crystals with F^- caused the luminescence spectrum composition to change for crystals of both series. The observed changes were most evident in the temperature range 260–120 K whereas the effect of doping on the luminescence spectrum composition was less noticeable as the temperature was reduced further to 10 K. The luminescence spectrum maximum for $PbWO_4$:F (K) shifted to the low-energy region (Fig. 1c); for $PbWO_4$:F (L), to the high-energy region compared with the pure sample (Fig. 1d). It should be noted that the temperature dependences of the spectral composition for the doped samples were similar although the spectral composition and shift of maxima for the pure crystals were different.

Measurements of luminescence spectra of undoped samples under identical conditions (T = 10 K, λ_{ex} = 115 nm) found that the emission intensity of the series L sample was 16% greater than that of the series K sample. Fluo-

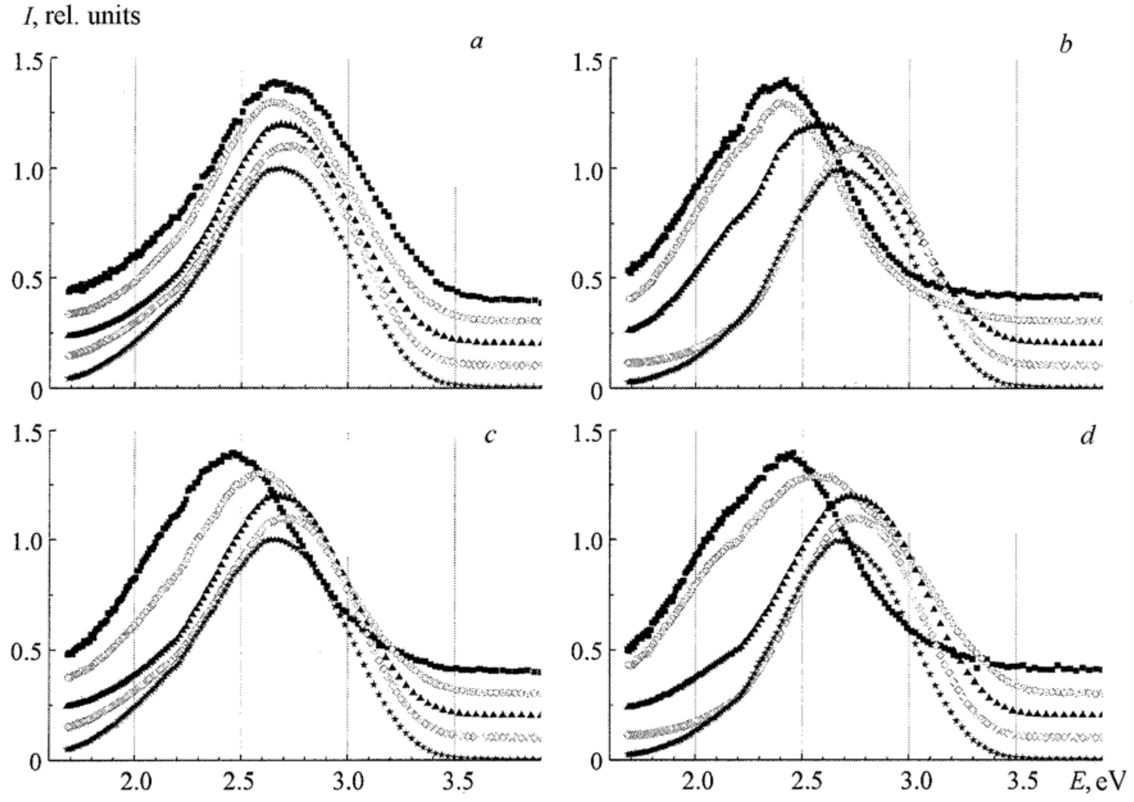

Fig. 1. Luminescence spectra of PbWO4 (a, b) and PbWO$_4$:F (c, d) crystals of series K (a, c) and L (b, d); T = 10 (★), 60 (◇), 120 (▲), 200 (○), and 260 K (■).

TABLE 1. Position of Luminescence Band Maxima (E_{max}) and Their Full-Width at Half-Maximum (FWHM) (in eV) for Samples at Various Temperatures

T, K	PbWO$_4$ (K)		PbWO$_4$ (L)		PbWO$_4$:F (K)		PbWO$_4$:F (L)	
	E_{max}	FWHM	E_{max}	FWHM	E_{max}	FWHM	E_{max}	FWHM
10	2.65	0.77	2.65	0.73	2.65	0.79	2.67	0.7
60	2.75	0.76	2.75	0.7	2.72	0.78	2.75	0.7
120	2.7	0.74	2.6	0.91	2.65	0.74	2.72	0.78
200	2.65	0.78	2.4	0.73	2.6	0.81	2.57	1.09
250	2.65	0.82	2.4	0.75	2.45	0.78	2.45	0.76

rine-doping increased the luminescence intensity of the series L samples by 22%. Similar measurements for the doped series K samples were not made.

Figure 2 shows luminescence intensities as functions of temperature. The intensities were obtained by integrating the luminescence signal over the whole energy range of the measured spectra (1.6–4.0 eV) at the given temperatures. The temperature dependence grew considerably in the range 300–140 K and reached a plateau as the temperature was decreased further upon excitation of luminescence in the region of the exciton peak (4.1 eV) (Fig. 2a, curve 3). This dependence was due to intracenter thermal quenching and could be described using the Mott formula [18]. The activation energy of the process (147 ± 11 meV) was calculated using this formula. It is noteworthy that only the fundamental luminescence band was present in the luminescence spectrum upon excitation at the exciton

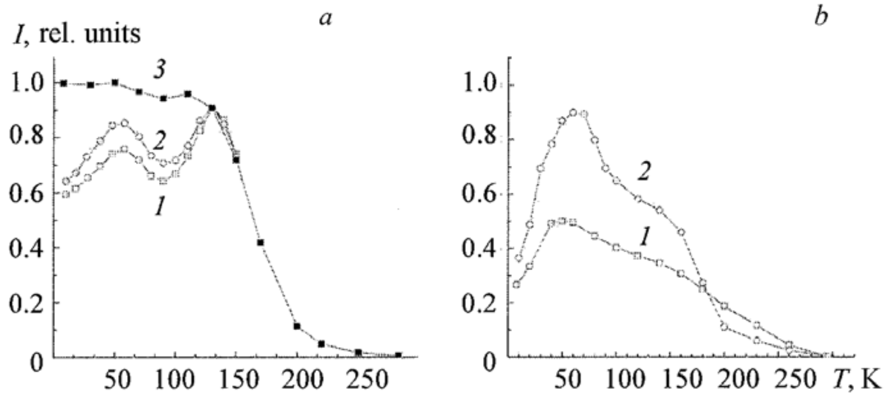

Fig. 2. Luminescence intensity of $PbWO_4$ (1, 3) and $PbWO_4$:F (2) crystals of series K (a) and L (b) as functions of temperature; E_{ex} = 13.8 (1, 2) and 4.1 (3) eV.

peak. The nature of the dependence became complicated as the excitation energy was increased. Two maxima appeared at 150 and 60 K. The luminescence intensity was noticeably weakened at low (10 K) temperatures. Fluorine-doping had practically no effect on the temperature dependence of the luminescence intensity. However, the luminescence intensity of $PbWO_4$:F was greater than that of the nominally pure crystal at 10–140 K.

The principal features of the integrated luminescence intensity as a function of temperature that were observed in series K crystals were also characteristic of series L (Fig. 2b). In fact, the peak at 60 K was also clearly visible whereas the peak at 150 K appeared as a shoulder. The decrease was more pronounced at low temperatures. The luminescence of the $PbWO_4$:F sample was also greater than that of $PbWO_4$ at 150–10 K.

Luminescence decay kinetics. Figure 3 shows luminescence decay kinetic curves of the studied crystals that were measured at E_{ex} = 13.8 eV with E_{lum} = 2.75 eV and T = 230 and 300 K. The function $I(t) = y_0 + A_1 \exp(-t/\tau_2) + A_2 \exp(-t/\tau_2)$, where τ_1 and τ_2 were the decay times of the fast and slow luminescence components and A_1 and A_2, the amplitudes of the corresponding components, was used to approximate the decay curves. Table 2 lists the resulting decay times for the various samples. The presence of two components may have been related to thermal luminescence quenching of $PbWO_4$ in the range 230–300 K. Both the blue luminescence band, which exhibited faster decay kinetics (units and tens of nanoseconds), and the green luminescence band, for which slower times were characteristic (>1 μs), contributed to the resulting curves [15, 19].

Faster decay kinetics were observed for the $PbWO_4$ (K) sample. This agreed with the luminescence spectrum composition of the sample, where the blue component dominated. The $PbWO_4$ (L) sample had slower decay kinetics, which also correlated with the luminescence spectrum, where the emission maximum corresponded to the position of the green band. Doping had opposite effects on the luminescence decay kinetics. The decay times increased for the series K samples (Fig. 3a and c) and decreased for the series L samples (Fig. 3b and d). The decay kinetics of the fluorine-doped samples were similar for the different series.

Thermoluminescence (TL). Figure 4 shows TL spectra of $PbWO_4$ and $PbWO_4$:F crystals. The samples were irradiated at 10 K by phonons of energy 11 eV for 5 min before the TL was measured. Spectra were recorded for E_{lum} = 2.5 eV (λ = 500 nm). This made it possible to observe TL peaks emitted through both the blue and green $PbWO_4$ bands.

According to the TL spectra, F had practically no effect on the traps existing in the series K crystal (Fig. 4a). Peaks at 50 and 104 K were present in spectra of both the fluorine-doped and nominally pure samples. Their positions and intensities did not change. An additional weak peak at 165 K was observed only in the TL spectrum of the F-doped sample. The peak at 50 K is usually associated with the electron center $(WO_4)^{3-}$ [20]. The peak at 104 K is ascribed to the complex center $(WO_4)^{3-}$–A^{3+} [21], where A^{3+} is a rare-earth dopant. The series K crystals contained an impurity of Y according to chemical analysis. Therefore, the TL peak and the presence of this impurity can be assumed to be related. Approximation of the two strong peaks using a first-order kinetic approach [22] enabled the ac-

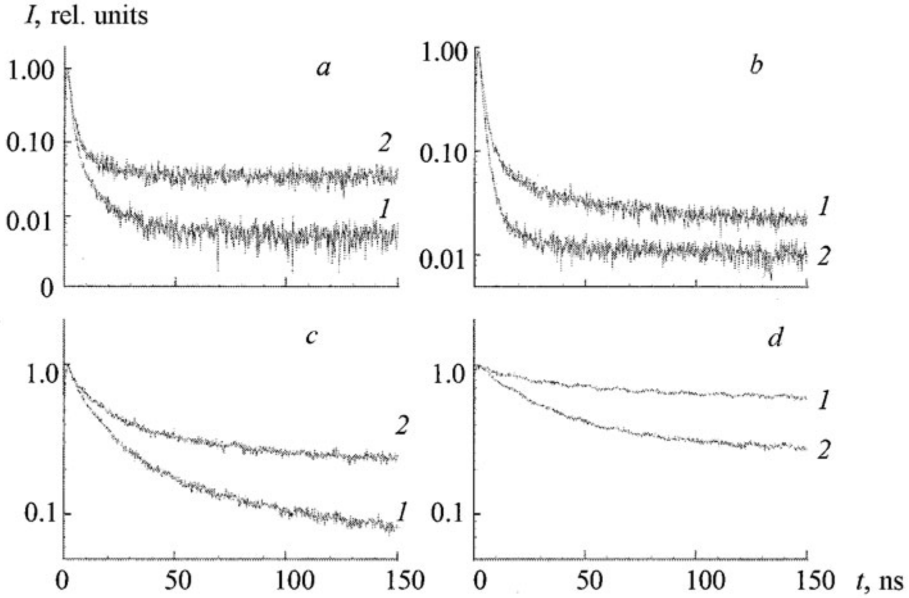

Fig. 3. Luminescence decay kinetics of $PbWO_4$ (a, b) and $PbWO_4$:F (c, d) crystals of series K (a, c) and L (b, d); T = 300 (a, b) and 230 (c, d) K.

TABLE 2. Luminescence Decay Times (ns) of Samples Obtained from an Approximation of the Sum of Two Exponents at 230 and 300 K

Time, ns	$PbWO_4$ (K)		$PbWO_4$ (L)		$PbWO_4$:F (K)		$PbWO_4$:F (L)	
	230 K	300 K	230 K	300 K	230 K	300 K	230 K	300 K
τ_1	11.3	2.9	25.6	2.5	13.2	1.8	23.3	1.7
τ_2	77.6	1.4	953.3	124.9	319.3	13.8	500.7	14.6

tivation energy of the traps responsible for the two strongest peaks to be determined as 65 and 129 meV. The weak peak at 165 K was observed only in the TL spectrum of the F-doped sample and could be associated with the presence of this element in the $PbWO_4$ structure.

The effect of F on the TL spectrum was more evident for series L crystals (Fig. 4b). A broad combination peak in the range 150–240 K was observed in the TL spectrum of $PbWO_4$ (L) in addition to the peaks that were observed for series K samples in the range 40–140 K. It was shown based on studies using EPR [23] that the peaks in this temperature range were due to the presence of oxygen vacancies in the $PbWO_4$ structure (Pb^+–V_O). It is noteworthy that the intensities of the peaks in this temperature range decreased substantially for $PbWO_4$:F (L). In fact, weak peaks with maxima at 162, 179, and 195 K were observed in the TL spectrum.

The experimental results indicated that nominally pure $PbWO_4$ crystals could have markedly different luminescent properties depending on the laboratory in which they were grown. This was evident in the luminescence spectrum composition, the decay kinetics, and the TL spectra of the studied crystals. The results as a whole led to the conclusion that structural defects in the form of oxygen vacancies were typical of $PbWO_4$ (L). Furthermore, rare-earth impurities were present in both series of crystals. This produced a TL peak at 104 K. Primarily fundamental luminescence without a noticeable contribution from luminescence at structural defects was observed for $PbWO_4$ (K) crystal. TL peaks in the range 120–300 K that were due to structural defects or a Mo impurity were also missing [20, 23]. It could be concluded that this sample had a low concentration of structural defects.

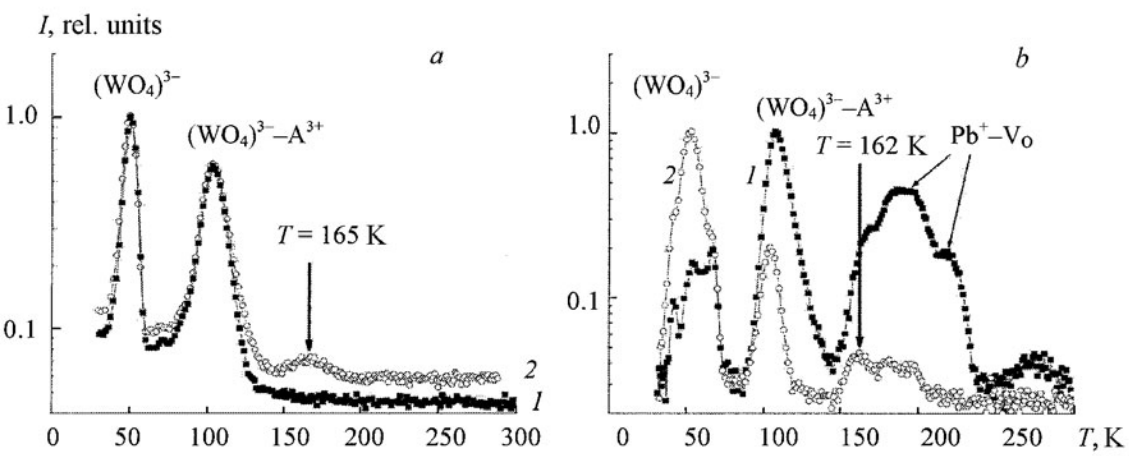

Fig. 4. Thermoluminescence spectra of $PbWO_4$ (1) and $PbWO_4$:F (2) of series K (a) and L (b).

It is noteworthy that a peak at 50 K was characteristic of most studied PWO crystals and was associated with electron capture at the regular oxyanion complex. The presence of such a trap could explain the decreased luminescence intensity in the range 60–10 K (Fig. 2) because traps are an additional relaxation channel for electrons excited into the conductivity band. A decrease of luminescence intensity at low temperature was not observed upon excitation at the exciton peak (curve 3). Excitons were directly produced whereas separated electron–hole pairs were not created by this excitation. The presence of a trap in $PbWO_4$ that was not associated with impurities or structural defects had a negative effect on the light output of scintillations at low temperatures. This was important in view of the potential use of this crystal in experiments on the recording of rare events at ultralow temperatures [3]. Fluorine-doping caused a significant change of luminescent properties in the studied crystals. The spectrum composition, decay kinetics, and TL curves of samples doped with F were similar.

Introducing F^- into the $PbWO_4$ structure presupposes replacement of O^{2-} ions in the WO_4^{2-} complex oxyanion because of the similar atomic radii (1.26 Å for O^{2-} and 1.19 Å for F^-) and identical electronic configurations. A center (WO_3F) with the same symmetry as the center of green emission $WO^{3+}F^-$ (F^+:V_O+e) could be formed as a result of the considerable hybridization of the F and O 2*p*-states in WO_4 [24]. If V_{Pb} vacancies were assumed to be present in the $PbWO_4$ matrix, then substitution of oxygen positions by F^- anions $F_O^\bullet$ would create an excess of positive charge in the oxygen sublattice. Electroneutrality in this instance would be achieved by forming dipolar complexes of the type $[2F_O^\bullet\text{-}V_{Pb}'']$ through cation vacancies V_{Pb}'' [7].

It was demonstrated previously [13] that the complex WO_3F was characterized by the appearance of an additional band in the green spectral region that overlapped considerably the luminescence band associated with the presence of structural defects. This band was observed as a shoulder with a maximum at 2.25 eV (~550 nm) on the main peak at 2.45 eV (~505 nm). The appearance of the WO_3F luminescence center for series K crystals led to a long-wavelength shift of the luminescence spectrum. The lack of TL peaks characteristic of crystals with oxygen vacancies confirmed that this emission center was present.

The intensity of TL peaks in the range 140–240 K for series L $PbWO_4$:F was noticeably less than for the undoped sample. Thus, the intensity of the high-temperature TL peaks decreased by 2.5 times relative to the peak at 104 K. The decrease in the amount of oxygen vacancies responsible for the TL peaks in the range 140–240 K could be due to the curing of crystal structure defects by forming WO_3F complexes. It could be said that the emission intensity increased in the temperature range 10–150 K in F-doped samples based on luminescence intensity data (Fig. 2b). An analogous effect was observed also for series K samples although it was less pronounced.

Based on the results, an increase of light output of scintillations could be expected for $PbWO_4$:F at low temperatures if this material was used in cryogenic phonon-scintillation detectors This hypothesis was also confirmed by the following observation. The output of scintillations as a function of temperature for series L $PbWO_4$ crystals was reported [25]. Its principal features were reproduced in the temperature dependence of the luminescence upon VUV excitation (Fig. 2b). This indicated that the principal physical processes affecting the change of crystal scintillation properties with temperature were active even upon excitation by VUV photons, the energy of which was substantially less than the operating energies of the scintillation crystals.

Conclusion. The effect of fluorine-doping on the luminescent properties of two series of $PbWO_4$ samples grown by the Czochralski method in different laboratories was studied. Undoped $PbWO_4$ crystals of the different series exhibit markedly differing luminescent properties. It was shown that this was associated with different concentrations of structural defects as oxygen vacancies in $PbWO_4$. The luminescence spectrum composition, decay kinetics, and TL curves for $PbWO_4$:F of both series were similar after doping. This was associated with the formation of WO_3F and $PbWO_4$:F complexes. In particular, it was shown that fluorine-doping decreased the concentration of oxygen vacancies and increased the luminescence intensity of $PbWO_4$.

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